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NAVAL CONSTRUCTION BATTALION CENTER

Port Hueneme, California 93043

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coatings on untreated concrete. Test specimens were exposed at Port Hueneme, Calif., and Key Biscayne, Fla. Efficacy of toxicants was determined by periodically weighing the specimens and the fouling organisms that became attached. Concrete prepared with an aggregate impregnated with a TBTO/creosote mixture demonstrated the best antifouling performance of those specimens exposed for more than 1 year. The two proprietary coatings and the concrete containing methoxychlor, TPTH, and cuprous oxide as dry additives have exhibited good antifouling properties but have been exposed for a shorter time. Also, the strength of concrete prepared using the toxicants was acceptable, and the corrosion rate of reinforcing rods did not increase. The concentration of organotin compounds was essentially unchanged in a concrete specimen exposed 6-1/2 years in seawater.

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Various toxic agents were investigated for their ability to prevent the attachment and growth of marine fouling organisms on concrete. Three methods of incorporating antifoulants into concrete were also studied. Porous aggregate was impregnated with creosote and bis-(tri-n-butyltin) oxide (TBTO) and then used in making the concrete. Cuprous oxide, tri-phenyltin hydroxide (TPTH), and 2-2-bis-(*p*-methoxyphenyl)-1,1,1-trichloroethane (methoxychlor) were used as dry additives. Two proprietary formulations were applied as coatings on untreated concrete. Test specimens were exposed at Port Hueneme, Calif., and Key Biscayne, Fla. Efficacy of toxicants was determined by periodically weighing the specimens and the fouling organisms that became attached. Concrete prepared with an aggregate impregnated with a TBTO/creosote mixture demonstrated the best antifouling performance of those specimens exposed for more than 1 year. The two proprietary coatings and the concrete containing methoxychlor, TPTH, and cuprous oxide as dry additives have exhibited good antifouling properties but have been exposed for a shorter time. Also, the strength of concrete prepared using the toxicants was acceptable, and the corrosion rate of reinforcing rods did not increase. The concentration of organotin compounds was essentially unchanged in a concrete specimen exposed 6-1/2 years in seawater.

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## INTRODUCTION

For centuries ships were "careened," or laid over on their sides, to remove fouling from their hulls and replace the materials then used as antifoulants. In 1495, the first dry dock was built for Henry VII at Portsmouth (Ref 1), and this method for removal of fouling has today replaced careening. Neither of those methods is presented as a possible way to keep an Ocean Thermal Energy Conversion (OTEC) facility free of fouling, but they are cited to present the difficulty man has encountered over the centuries in ridding his ocean-going vessels of accumulating biomass.

This investigation was directed at the production of concrete which will resist the attachment of marine fouling organisms. Such a concrete is needed to form or line the hull and cold water pipe of the proposed OTEC structure. Growth of marine fouling organisms on a floating structure can add significantly to its weight, thus reducing freeboard. For instance, biomass has accumulated to 25 lb/ft<sup>2</sup> on buoys in less than 3 years (Ref 2, p. 17). The cold water OTEC pipe, when fouled, would provide a reservoir of adult organisms (Ref 2, pp 11-14) that could discharge eggs or larvae directly into the heat exchanger surfaces. Adult members of an established fouling community would be sloughed off in time, causing clogging of screens. Broken pieces of calcareous shells of adults carried in the cold water intake would present further problems. The Civil Engineering Laboratory (CEL) has undertaken an investigation of methods to make concrete that resists the attachment of marine fouling organisms.

Ten years ago at CEL, James S. Muraoka prepared concrete incorporating toxic chemicals as antifoulants (Ref 4,5). This method of incorporating oily toxicants into the concrete was patented (Ref 6). In this method, a porous, expanded shale aggregate was impregnated with such toxicants as bis-(tri-*n*-butyltin) oxide (TBTO) and creosote. Concrete was then prepared from the treated aggregate, cement, and water. Toxicants other than TBTO and creosote were evaluated. These included pentachlorophenol, quaternary amines, malachite green base, malachite green oxalate, tributyltin fluoride, nicotine, and copper naphthenate. Muraoka concluded that an impregnant consisting of a mixture of creosote and TBTO (3:1 by volume) yielded concrete having the best antifouling properties (Ref 5). Because the interest at that time was primarily in antifouling properties, sand was not used in the concrete mix design. As a result, the compressive strength of the resulting product was lower than that of standard concrete. With the advent of the OTEC concept, an antifouling concrete with a higher compressive strength was required. The work described in this document is directed toward that objective.

## ANTIFOULING MATERIALS

Three methods of preparing antifouling concrete were investigated.

In the first method, a porous expanded shale aggregate was impregnated with liquid toxicants, and concrete was prepared in an otherwise normal manner. In another method, dry toxicants were added directly to the concrete mix. The toxicants employed as additives in this investigation were organotin compounds, creosote, cuprous oxide, and a chlorinated organic insecticide. These were evaluated alone or in certain combinations. The third method involved the application of a special coating to otherwise untreated concrete. Two antifouling coating systems were investigated, both containing organotin toxicants.

All of the materials investigated were incorporated into or were applied to 3-in.-diam x 6-in.-long concrete cylinders. The test cylinders were then exposed in the ocean at Port Hueneme, Calif., and Key Biscayne, Fla. The cylindrical shape was chosen so that compressive strength determinations could be made according to methods stipulated by the American Society for Testing and Materials (ASTM) (Ref 7). The antifouling properties of candidate concretes were measured by periodically weighing the specimens and attached fouling organisms. This resulting weight increase was compared with that of untreated controls which were similarly weighed to determine, by difference, the weight of accumulated biomass.

### Treated Aggregate

Liquid toxicants were impregnated into porous, expanded shale aggregate to establish a reservoir of these antifouling chemicals in the finished concrete. The aggregate was first dried at 110°C to remove moisture. A list of the materials used in this work is presented in Table 1.

Two methods of incorporating liquid toxicant into the aggregate were evaluated.

In the first, the aggregate was placed in a container, covered with creosote, and allowed to stand 48 hours. The creosote was poured off, and the treated aggregate was allowed to drain, then spread on newspapers overnight to remove excess creosote from the surface. Using this method, an uptake of creosote equal to 16.0% of the weight of the aggregate was realized.

Vacuum impregnation of the aggregate was then evaluated. In this procedure, the dried aggregate was placed in a vacuum chamber from which the air was removed. Creosote was admitted until the aggregate was covered, whereupon air was allowed to return the pressure in the chamber to atmospheric, forcing the creosote into the pores of the aggregate. After a 1- to 2-hour soaking time, the creosote was removed and the aggregate dried in the same manner as before. This impregnation method yielded a creosote uptake of 36.8% of the weight of the aggregate and was used in all subsequent work. Toxicants used to treat aggregate by the vacuum impregnation method were bis-(tri-*n*-butyltin) oxide (TBTO) alone and 60 parts TBTO mixed with 40 parts creosote by weight (Table 2). The average uptake of TBTO alone by the aggregate was 40%, while the average uptake of the TBTO/creosote mixture was 38%.

Before incorporation into a concrete, the surface of the treated aggregate was rinsed to ensure a good bond between it and the cement paste. Several methods of rinsing were evaluated, including steam, hot water, cold water, trisodium phosphate solution, mineral spirits, and acetone-water solution. Based on the compressive strength of concrete made from aggregates rinsed by these methods, the acetone:water ratio of 90:10 by volume was selected as the method of choice and was used for all treated aggregate employed in this investigation. In this procedure, 3 kg of aggregate are shaken with 1 kg of solvent for 1/2 to 1 minute. The solvent is immediately decanted from the aggregate which is again spread on newspaper or burlap to complete solvent evaporation. The finished aggregate is either used immediately or stored in sealed containers until ready for use.

#### Dry Toxicants in Cement Mix

For some of the tests, cuprous oxide ( $Cu_2O$ ) was added as a dry ingredient to the concrete mix. It was added by itself and in combination with TBTO, but was not impregnated into aggregate.

Methoxychlor, a chloro-organic compound, was also investigated. It, too, was added to the concrete mixture as a dry ingredient, both by itself and in combination with triphenyltin hydroxide (TPTH) and  $Cu_2O$ .

#### Antifouling Coatings

Two types of coatings for concrete cylinders were tested. With one, an organotin-polysiloxane polymer coating was applied by brush to untreated cylinders. With the other, a proprietary TBTO-impregnated elastomer was wrapped around and cemented to untreated cylinders.

### TESTING PROCEDURES

#### Preparation of Concrete Specimens

The amounts of the constituents of the concrete are given in their order of addition:

40.3 lb expanded shale aggregate (weight before impregnation)

62.2 lb sand

~21 lb water

47.0 lb Type II portland cement

The wet concrete was mixed 3 minutes in a 1-3/4 ft<sup>3</sup>-capacity pan-type mixer. The slump was then measured; adjustments were made, if necessary, by adding more water. The finished wet concrete was then placed into 3-in.-diam x 6-in.-long cylinder-shaped molds to half fill them. The molds were placed on a vibrating table for several minutes and then filled to the top. The vibration was resumed and continued for several more minutes, after which the tops of the molds were finished with a trowel. The specimens were then placed in a 100% relative humidity

(fog) room overnight. The next morning, the molds were removed, and the specimens returned to the fog room to cure for 28 days.

The mix design for the concrete was not changed when various amounts of dry toxicants were added. In one case, pea gravel was substituted for expanded shale aggregate to compare compressive strengths. This was done in such a way that the same volume of pea gravel was used as was occupied by 40.3 pounds of expanded shale aggregate; thus, 79.5 pounds of pea gravel were used in this mix.

The first specimens for testing were formed in 1977. Antifouling concrete containing aggregate impregnated with TBTO and creosote (60:40 by weight) was prepared (Ref 8,9). A set of control specimens was also prepared, using the same mix design, but with untreated aggregate. This first set of specimens is designated as U1, U2, C1, and C2 (see Table 2). Twelve replicates of each were placed in the channel at Port Hueneme harbor for 173 days. At periodic intervals during exposure, ratings were made of the types and approximate numbers of the types of fouling attached to the specimens. When the specimens were removed from the ocean they were weighed, dried, and scraped free of the accumulated biofouling. At this time, it was decided that the more objective gravimetric ratings gave a better indication of the antifouling performance than the more subjective visual observations done periodically during the exposure (see Table 3). Subsequently, weighing was done on all specimens exposed.

The remaining sets (2 through 5 of Table 2) were exposed both at Port Hueneme and from docks at the University of Miami in Biscayne Bay. Set no. 2 contained four types of concrete: one duplicated the TBTO/creosote mixture from the first set, one contained TBTO alone as a toxicant, and the other two were controls: one prepared with pea gravel to compare its compressive strength with concrete made with treated and untreated expanded shale aggregate. Ten replicate specimens of each type of concrete were exposed at both sites.

Cuprous oxide ( $Cu_2O$ ) was employed in set no. 3, both by itself and in combination with TBTO. The  $Cu_2O$  was simply added to the concrete mix and wasn't impregnated into aggregate. As in the second set, TBTO was used by itself, but in this set the concentration of TBTO was halved in all cases by substituting an equal amount of pea gravel for treated aggregate. It was felt that this would both reduce the cost and increase the compressive strength of the resulting concrete. Five replicates of this set were deployed at both sites. Also, five replicates of each member of the third set were placed in the submarine harbor at Key West, Fla. to determine the resistance of these concretes to attack by pholads. No weighings were done at Key West, but visual inspections were made and photographs taken.

The effect of the chloro-organic compound methoxychlor was investigated in set no. 4. In two members of this set, the toxicants added to the concrete were dry powders and were incorporated into the concrete mix by simple addition. Methoxychlor was incorporated into the concrete by itself and in combination with triphenyltin hydroxide (TPTH) and cuprous oxide. One member of this set was not added to the concrete but consisted of an organotin-polysiloxane polymer coating brush-applied to otherwise untreated cylinders. Polymerization took place when the substance was exposed to atmospheric moisture. The coverage was deter-

mined by weighing to be 9.05 mg/cm<sup>2</sup>. Controls from a previous untreated set were deployed at both sites along with five replicates of each candidate antifouling concrete.

Set no. 5 was prepared by wrapping and cementing a proprietary TBTO-impregnated elastomer about untreated cylinders. No controls were prepared in this case. Five samples were placed at each of the exposure sites.

#### Ocean Exposure

Design Setup. Candidate antifouling concrete specimens were prepared as 3-in.-diam x 6-in.-long cylinders so that compressive strengths could be determined (Ref 10). To prepare the cylinders for exposure in the ocean, two small rectangular connector rings were threaded onto each of two nylon cable ties. One cable tie was tightly fastened about the circumference at each end of a cylinder with the connector rings on each tie located 180 degrees apart and in line with one another at opposite ends of the cylinder (see Figure 1). Finally, an identifying number was melted into each nylon cable tie with a soldering pencil. Cylinders prepared for exposure by this method were attached to one another by placing still other nylon straps through the connector rings of adjacent specimens loosely enough that they hung at a distance from each other, forming what could be described as rungs of a ladder. Such groups of cylinders, each suspended horizontally, were hung from docks so that the topmost cylinder was beneath water during an extreme low tide. Four cylinders were typically deployed in each group. At Port Hueneme, their position relative to one another in the group was changed periodically when they were weighed to eliminate the effect of the depth variable. Also, the place from which each group was suspended from the dock was changed to prevent the entrance of effects of other variables into the results.

Sites. Port Hueneme was chosen as an exposure site because the Civil Engineering Laboratory is close by. Specimens were exposed in the channel to this deep-water port where a good exchange of water from the ocean is made daily - semi-diurnal tides range from 4 to 9 feet. The temperature extremes of the surface water range from 9° to 22°C (Ref 11).

Key Biscayne, Fla., was chosen as a second exposure site because of its warmer water and the availability of qualified personnel at the University of Miami to monitor the antifouling properties of the specimens. At Key Biscayne, the specimens were exposed in shallower water than at Port Hueneme, but they were placed in a location where a 5-knot current flowed about them. It is felt that this relatively high flow rate would at least approach simulation of flow conditions one might encounter in the cold water pipe of an OTEC platform. The tidal extremes at Key Biscayne are less than 2 feet, and the water temperature extremes in 1977 ranged from 15° to 32°C (Ref 12).

#### Corrosion of Simulated Reinforcing Rod

The effect on the corrosion rate of simulated reinforcing rods by toxicants employed in antifouling concrete set no. 2 and 3 was investigated. One cylinder from each set was crushed and the pieces placed in

a 5-liter plastic bucket which also contained one intact cylinder prepared with two 6d nails in its interior. Ten weighed 6d nails were added to each bucket, and seawater was allowed to trickle into it so that it constantly overflowed. The seawater addition was from a sufficient height that splashing and consequent aeration of the bucket's contents occurred. The nails were periodically removed, ultrasonically cleaned, rinsed with demineralized water, dried, and weighed to determine weight loss. When the experiment was terminated, the intact specimens were crushed and the nails examined for the presence of corrosion products.

In a parallel experiment, cylinders prepared with 6d nails both at the center and 1 cm from the wall were exposed to partial immersion. Here, each of the above cylinders was placed vertically in a 2-in.-deep pan through which seawater continually flowed.

The test cylinders containing the nails were exposed in a partial immersion test for 446 days. Inspections were periodically made to determine whether possible oxidation of the steel caused cracking or spalling of the concrete.

#### Biotoxicity Tests

Two experiments were designed to determine the relative toxicity of certain of the toxicants employed as antifoulants. Fertilized eggs of the red abalone, Haliotis rufescens, were used as test organisms in the first experiment. Approximately 10,000 eggs were introduced into each of two 50-liter plastic containers, one containing a control concrete specimen, and the second a specimen prepared using TBTO-impregnated aggregate. The next day the development of the eggs in both containers had terminated at the multicellular level. These concrete specimens, as well as others, were then placed in flowing seawater and allowed to leach for 102 days. The experiment was then repeated using 17-liter containers with ~5,000 fertilized eggs in each.

A second experiment was designed to measure the relative concentrations of various toxicants required to kill larvae of H. rufescens. In this experiment, treated and control concrete cylinders were leached for 12 days then placed in plastic pails containing 4.3 liters of seawater that was filtered to 1 micron and sterilized with ultraviolet radiation. The cylinders were immersed for 2, 20, and 200 minutes in such a way that no part of the cylinder contacted the container. The larvae were then introduced and the effect of the three concentrations of various toxicants was monitored by periodic microscopic observation of the larval development.

## FINDINGS

#### Antifouling Properties

Set no. 1, TBTO/Creosote Mixture. After 173 days of exposure, the concrete prepared with the TBTO/creosote-impregnated aggregate accumulated 44% as much biofouling as the untreated control. Table 3 presents these data and summarizes the final subjective evaluations made after 163 days of exposure (10 days before removal of the cylinders).

Set no. 2, TBTO Alone and TBTO/Creosote Mixture. In this set, TBTO alone did not perform as well as the TBTO/creosote mixture (see Table 4). Cylinders prepared with TBTO alone accumulated 83% as much biofouling as the control after 596 days at Port Hueneme, compared with 52% for the TBTO/creosote mixture (Figure 2). At Key Biscayne, biofouling was 96% of the control for TBTO alone, and 59% for TBTO/creosote after 524 days (Figure 3).

Set no. 3, TBTO Alone, Cuprous Oxide Alone, and TBTO/Cuprous Oxide Mixture. TBTO was employed at half the concentration used in set no. 2, both alone and combined with cuprous oxide. Cuprous oxide alone was also evaluated and found to have no effect at the concentration used. At Port Hueneme the weight increases after 153 days due to biofouling of members of this set compared to the control were: TBTO alone, 81%; TBTO/Cu<sub>2</sub>O, 69%, and Cu<sub>2</sub>O only, 93% (Figure 4). Data from Key Biscayne after 283 days showed the same trend: TBTO alone, 92%, TBTO/Cu<sub>2</sub>O, 93%, and Cu<sub>2</sub>O only, 116% (Figure 5). The specimens in this set exposed at Key West have not shown any signs of attack by pholads. The cylinders are suspended a few feet from a 67-year-old concrete pier which exhibits evidence of pholad activity.

Set no. 4, Methoxychlor Alone, Methoxychlor/Cuprous Oxide/TPTH Mixture, and Organotin Coating. This set was prepared to investigate the antifouling properties of two basic toxicants: methoxychlor and a proprietary organotin coating. Methoxychlor was employed alone and in combination with TPTH and Cu<sub>2</sub>O. At Port Hueneme, the weight increase of members of this set versus an untreated control after 153 days was: methoxychlor alone, 124%, methoxychlor with TPTH and Cu<sub>2</sub>O, 65%, and organotin coating, 42% (Figure 3). At Key Biscayne, the results for replicates of this set after 139 days were: methoxychlor, 99%, methoxychlor with TPTH and Cu<sub>2</sub>O, 90%, and organotin coating, 66% (Figure 6).

Set no. 5, Organotin-Impregnated Rubber Cover. The cylinders covered with the organotin-impregnated rubber accumulated an average of 15.8 gm of biofouling after 60 days in Port Hueneme. No data are available at this time on the replicates exposed in Key Biscayne.

#### Compressive Strength

Various solvents for washing aggregate impregnated with TBTO/creosote (60/40) were tested. The prepared specimens had 7-day compressive strengths shown below when tested according to ASTM Method C-873-77T (Ref 10):

<u>Wash Solvent</u>	<u>Compressive Strength (psig)</u>
None (control)	3,920
Acetone:water (90:10)	3,660
None	3,190
Mineral spirits	2,960
Trisodium phosphate solution	2,720

The first set of concrete cylinders was prepared with treated aggregate rinsed in the acetone-water solution, which had been selected as the best solvent. After 6 months of exposure in Port Hueneme, the compressive strength of treated cylinders was 4,200 psi, while that of untreated controls was 5,140 psi.

The 28-day compressive strengths of cylinders from set no. 2 are compared below to those duplicates from this set exposed 9-months in the ocean:

<u>Treatment</u>	<u>Designation</u>	<u>28 Days</u>	<u>9 Months</u>
Control	UT	5,710	6,710
Pea gravel	PG	6,080	
TBTO	TB	4,840	6,090
TBTO/creosote	KR	3,200	3,840

The 9-month exposure measurements were for the Key Biscayne cylinders.

The third set of concrete specimens had 28-day compressive strengths as follows:

<u>Treatment</u>	<u>Designation</u>	<u>28-Day Compressive Strength (psig)</u>
Control	V4	4,660
TBTO	V1	6,010
TBTO/Cu <sub>2</sub> O	V2	5,360
Cu <sub>2</sub> O	V3	5,330

The compressive strength of the fourth set of cylinders was not determined.

#### Corrosion of Simulated Reinforcing Rod

The loss in weight of nails used to simulate reinforcing rod versus time is presented in Figure 7. All of the candidate antifouling chemicals evaluated in this test inhibited rather than accelerated the corrosion of the steel except cuprous oxide, and that only when employed by itself. The presence of TBTO, creosote, or a mixture of these compounds caused a significant reduction in the rate at which the steel corroded.

After 446 days exposure, the percentage of weight loss of the 10 replicate nails exposed to the various concretes was:

<u>Treatment</u>	<u>Weight Loss (%)</u>
Control	29.7, 29.6, 28.9
$\text{Cu}_2\text{O}$	28.3
TBTO and $\text{Cu}_2\text{O}$	23.0
TBTO	22.2, 20.2
TBTO/creosote	17.9

In Figure 7, the results from those treatments which were duplicated are averaged.

None of the cylinders prepared with nails placed at the center and 1 cm from the surface showed signs of cracking or spalling after 445 days. These cylinders were exposed so that the bottom one-third was immersed in continually flowing seawater, while the top two-thirds of the cylinder was in air to simulate the hull of a concrete vessel.

#### Biotoxicity

Two experiments were performed to evaluate biotoxicity; the first using fertilized eggs of the red abalone *H. rufescens*, and the second using larvae of the same organism.

In the initial part of the first experiment, it was found that an untreated control specimen caused cessation of development and death of fertilized eggs, so specimens were leached in flowing seawater for 102 days and the test repeated. Concrete cylinders prepared using TBTO, TBTO/creosote, TBTO/ $\text{Cu}_2\text{O}$ ,  $\text{Cu}_2\text{O}$ , and an untreated control were evaluated.

Approximately 5,000 fertilized eggs were placed in 17 liters of seawater in a polyethylene bucket, into which a test cylinder was then placed. After 18 hours, the development of the eggs was observed microscopically. All eggs in buckets with a treated specimen had ceased development, while eggs in seawater with a control specimen were developing normally. Some differences were observed in the stages to which development progressed before death and in the morphology of the organism, depending on the toxicant present (Ref 13), but all the toxicants did cause cessation of development at some stage.

The second experiment used the larvae of *H. rufescens* as the test organism. In this test three toxicant systems were evaluated at three different concentrations. Cylinders prepared using TBTO alone, a TBTO/creosote mixture, and a methoxychlor/TPTH/ $\text{Cu}_2\text{O}$  mixture were exposed to seawater in polyethylene pails containing 4.3 liters of seawater for 2, 20, and 200 minutes. They were previously leached for 12 days in flowing seawater to establish a steady-state diffusion rate. An untreated control was similarly employed. Approximately 1,000 larvae were added to each pail, and the progress of development was observed periodically through a microscope. Table 5 shows the effect of the various toxicants at each concentration as a function of time. Differences in the morphology of the larvae exposed to the different toxicants were again observed. It is interesting to note that methoxychlor, a DDT analog, produced effects on the larvae more slowly than did other toxicants.

Generally, the TBTO and TBTO/creosote caused necrosis, followed eventually by cellular disassociation. This effect is similar morphologically to that of elevated temperature (Ref 14). Methoxychlor caused abnormalities in developing body parts. After 32 hours, death occurred in all cases where toxicant was present. The development of larvae in pails which contained the duplicate set of controls continued normally for 7 days, after which the experiment was terminated.

## DISCUSSION

### Cost of Materials

Toxicant cost varies greatly from one system to another. Because an OTEC plant will use a large amount of concrete, the cost of the material chosen as an antifoulant will be a major consideration. A comparison of the raw material cost of the various toxicants employed in this investigation is given below in 1979 dollars:

<u>Treatment</u>	<u>Cost</u>
TBTO (\$/lb)	6.55
Creosote (\$/lb)	0.10
Cu <sub>2</sub> O (\$/lb)	1.30
Methoxychlor (\$/lb)	1.40
TPTH (\$/lb)	8.16
Nofoul rubber, 0.08 in.-thick (\$/ft <sup>2</sup> )	4.85
Organotin polymer (\$/gal)	50.00

One of the better toxicant systems employs 60% TBTO and 40% creosote impregnated into aggregate. Because of the high cost of TBTO relative to creosote, concrete containing a higher ratio of creosote to TBTO should be considered. It is worth noting that 100% TBTO is a less effective antifoulant than the TBTO/creosote mixture employed. The immediate conclusion is that the creosote provides antifouling properties not present in TBTO alone. It has been observed, however, that concrete specimens prepared with TBTO/creosote and left in a fog room for a few months become covered with a layer of toxicant that has oozed from the concrete. This phenomenon is not observed in specimens prepared using TBTO alone. It may be that the creosote acts as a vehicle for the TBTO, allowing it to move more readily through the concrete to the surface where it is effective as an antifoulant. This phenomenon is considered of great importance (Ref 15). If this is the case, other solvents may be even more effective.

The cost of toxicant in concrete prepared using only TBTO at 8%, based on the weight of the other constituents, is \$65/ft<sup>3</sup>. When TBTO/creosote is used at 60/40, the cost is \$40/ft<sup>3</sup>. This is significant both from the standpoint of reducing cost and from the standpoint of better antifouling properties. Further dilution of TBTO by creosote

would reduce the cost still more and could yield a better product. If the treated concrete is spread 0.5-inch thick over standard concrete (as it might be in an OTEC application) the toxicant cost is \$1.64/ft<sup>2</sup> if 8% of the concrete weight is TBT0/creosote.

The set of specimens prepared using methoxychlor/TPTH/Cu<sub>2</sub>O appears to possess good antifouling properties. At the concentrations used, the cost of toxicants in this set is \$63.40/ft<sup>3</sup>. This amounts to \$2.64/ft<sup>2</sup> when applied at a 0.5-inch thickness.

Two sets of cylinders were evaluated where a coating was placed on otherwise untreated concrete. Both seem effective as antifoulants. The first of these is a brush-applied organotin polymer which would, at the level employed in this experiment, cost \$0.10/ft<sup>2</sup>. It should be able to be applied in a heavier coating, perhaps by repetitive applications<sup>®</sup>. The second is a proprietary organotin-impregnated elastomer (Nofoul rubber). The cost of the materials and adhesives in this system is \$4.85/ft<sup>2</sup>. A thickness of 0.08 inch is assumed. The manufacturer produces sheets having greater thicknesses at higher costs.

#### Ocean Site Effects

Differences in the rate of attachment and growth of fouling organisms at the two sites were expected. Figures 2 through 6 show these differences. As a general rule, the first members of the fouling community observed on specimens at Key Biscayne were algae. Barnacles soon followed, eventually covering the specimen completely. At Port Hueneme, the sequence of attachment of fouling organisms was slower so that many different organisms would be found at one time on specimens where there was attachment.

Each time a set of treated specimens was exposed, a set of controls was exposed with it. The importance of this cannot be overstated. For example, the number of larvae of barnacles in seawater varies tremendously at different times throughout the year and reaches maxima at different times in different years (Ref 2, Chap. 5). Similarly, one is cautioned not to compare weight increases of specimens exposed at one site versus those exposed at another. The only way an evaluation of the effectiveness of a given toxicant can be made is by comparing the weight increase of specimens prepared with it versus those of control specimens exposed at the same time in the same place.

#### Objective Ratings

Table 3 shows one subjective rating made on the first batch of specimens, and the final objective rating made on the same specimens. The objective gravimetric method yields a more easily handled numerical rating that can readily be plotted (Figures 2 through 6). Further, different people using this method will arrive at the same figure.

Subjective rating is highly dependent upon the rater's judgment, which varies from person to person and from day to day. For these reasons, the objective weighing method of evaluation was employed in this experiment. No major differences in types of fouling have been noted between treated specimens and untreated controls - only amounts.

### Persistence of Organotins

A test to determine the life of an organotin in a seawater environment was performed. A concrete panel made with TBTO-impregnated aggregate, cement, and water was prepared by Muraoka in May 1970, and placed in the harbor at Port Hueneme (Ref 4). In November 1977, it was removed from the harbor and cut with a concrete saw so that representative samples of both the interior and the outermost 3 mm were obtained. A portion of the sample from the interior was extracted three times with acetone and four times with benzene. Duplicates of these samples and a control which contained a known amount of TBTO were analyzed for tin by x-ray fluorescence spectroscopy. The analytical results were:

<u>Item</u>	<u>Location</u>	<u>Tin (%)</u>
Exposed sample	exterior	1.47, 1.35
	interior	1.52, 1.52
	interior (extracted)	0.24, 0.22
Control standard		4.75, 4.85

The standard was known to contain 14.8% tin, so the results should be raised about threefold. It will be seen, however, that the amount of tin found at the exterior of the specimen is nearly the same as that found at its interior. Furthermore, most of the tin present in the specimen was extractable in organic solvents; thus, it had not degraded to inorganic tin compounds during the 6-1/2 years the sample was in the ocean. It can be concluded that TBTO remains bound in an organic molecule for long periods of time in the antifouling concrete exposed in seawater.

### SUMMARY OF RESULTS

#### Antifouling Properties

Two systems employing toxicants as additives to concrete were found to have significant antifouling properties. The first of these employed porous aggregate impregnated with a mixture of bis-(tri-n-butyltin) oxide (TBTO) and creosote. The second employed triphenyltin hydroxide (TPTH), cuprous oxide, and methoxychlor, all of which were added to the concrete mix as dry powders.

Both systems in which concrete was coated with toxicants exhibited good antifouling abilities. One consisted of a liquid organotin-polysiloxane coating brush-applied and allowed to cure by reaction with moisture in the air. With the second a sheet of rubber impregnated with TBTO was secured to the concrete by an adhesive.

#### Toxicant Effects

Compressive strength tests of concrete which contained antifoulant chemicals were also conducted. Results indicated that a sufficiently strong antifouling concrete could be prepared.

Investigations into the effect of the antifouling additives on the corrosion of reinforcing rods were performed. These tests indicated that those additives which proved efficacious retarded the corrosion of steel reinforcement.

Biotoxicity tests revealed that the toxicants were lethal to test organisms in very small doses. Organotin (as TBTO) was found to remain bound in an organic molecule in concrete for 6½ years in the ocean.

#### Costs

The cost of the toxicants employed is significant and will be a criterion in the selection of an antifouling system. The coating system consisting of the organotin-polysiloxane polymer is the least expensive, but insufficient data have been gathered to allow an estimation of its useful lifetime. At the coverage rate used in this investigation, the cost of this product was \$0.10/ft<sup>2</sup>.

The TBTO-impregnated rubber is higher in cost - cost increasing with increasing thickness. The 0.08-in.-thick sheet of this rubber product costs \$4.85/ft<sup>2</sup> for material and adhesives. A 1-in.-thick panel has remained totally fouling free in Biscayne Bay for 7 years, according to the manufacturer. A 0.5-in.-thick sheet has an estimated antifouling life of 25 years in tropical waters.

The cost of the two toxicant-bearing concretes described in this investigation depends upon the thickness of the treated concrete layer applied over the concrete structure. Assuming a 0.5-in. layer, the cost of the toxicant in the TBTO-creosote system is \$1.64/ft<sup>2</sup>. This could be reduced (and perhaps an even better concrete prepared) by reducing the ratio of TBTO to creosote. The system employing TPTH, cuprous oxide, and methoxychlor carries a toxicant cost of \$2.64/ft<sup>2</sup> when applied 0.5-in. thick. Insufficient data are available to make it possible to speculate on whether this cost could be reduced significantly without losing the antifouling properties of the concrete.

#### CONCLUSIONS

Various candidate antifouling concretes were prepared and exposed in the ocean at two sites. The effects of the chemicals used as anti-foulants were investigated. Toxicity tests of the antifoulants on specific organisms were performed, and their effect on reinforcing rod was investigated. Results of these investigations are summarized as follows:

1. Concrete into which toxicants were incorporated accumulated, at best, about half as much fouling as untreated controls. This ability to resist fouling is insufficient for practical use in the present form. Two systems in which toxicant-bearing coatings were applied to concrete show promise, but they have been under investigation for only a short time.
2. A mixture of TBTO and creosote (60:40 by weight) impregnated into aggregate from which concrete is made yielded a product with better antifouling properties than concrete prepared from aggregate impregnated with TBTO only. This is highly significant from a cost standpoint.

3. Concrete prepared using TBTO-impregnated aggregate and cuprous oxide possesses some antifouling properties. Cuprous oxide, when employed as the sole antifoulant (at 2% by weight of the total) did not provide antifouling properties.
4. Concrete containing methoxychlor, TPTH, and cuprous oxide demonstrated antifouling properties, while concrete prepared using methoxychlor alone exhibited none. These specimens have been exposed for only five months.
5. A proprietary organotin-polysiloxane polymer coating was under evaluation for 5 months. Preliminary data indicate that this coating prevents fouling and is low in cost.
6. A proprietary organotin-impregnated rubber sheeting demonstrated excellent antifouling properties for 2 months at Port Hueneme. Its cost is significantly greater than that of the organotin-polysiloxane coating both for materials and application.
7. The cost of the various antifouling concretes varies considerably and will be an important consideration in the selection of one system versus another.
8. Structurally sound concrete can be prepared using aggregate impregnated with the antifoulants employed in this investigation. The compressive strength of these concretes was greater after 9 months of ocean exposure than after the 28-day curing period.
9. All of the candidate antifoulants considered for possible use caused a reduction in the corrosion rate of simulated steel reinforcing rods.
10. Some of the candidate antifouling concretes were exposed in an area of the ocean where rock-boring clams (pholads) are known to exist. In the 1-1/2 years this test has been under way, none of these concretes (including the untreated controls) has shown evidence of attack by the pholads.
11. Biotoxicity tests were performed with certain of the toxicants on the eggs and larvae of the red abalone Haliotis rufescens. All of the toxicants proved to be fatal to this organism at all concentrations tested.
12. A TBTO-containing antifouling concrete panel prepared in an earlier investigation and exposed in the ocean 6-1/2 years had a tin concentration essentially the same at both the surface and the interior. The tin at the interior was shown to still exist in an organic molecule.

#### RECOMMENDATIONS

Concrete made with aggregate impregnated with a TBTO/creosote mixture exhibits antifouling properties after 1-1/2 years of exposure. The ratio of TBTO to creosote might well be lowered to reduce the cost of the impregnant. Because it has been shown that this mixture of impregnants displays a latent period before the best antifouling properties are realized, this effect of time should be considered. If it is determined that creosote should not be used in concrete for an OTEC structure, other hydrocarbon vehicles for TBTO should be investigated.

After 5 months exposure, concrete containing a methoxychlor/TPTH/Cu<sub>2</sub>O mixture exhibits excellent antifouling properties. The exposure of these specimens will continue, and the efficacy of this concrete should be determined after a longer exposure.

The two proprietary coatings exhibit excellent antifouling properties, but these also have been under test for only 5 months. Because the organotin polysiloxane coating is the least expensive of any of the antifouling systems investigated, exposure should continue.

Alternatives to the use of any antifouling chemicals in the concrete exist. The hull could be scraped periodically by divers. Cold water pipes could be constructed of relatively inexpensive polymeric materials. They could be replaced as necessary, and the fouled pipes brought to a convenient place for scraping. These alternatives should be investigated to determine the most manpower- and cost-effective methods of controlling fouling of an OTEC facility.

#### ACKNOWLEDGMENTS

Until his death in 1978, Dr. Harold Vind, who originally worked on this project at CEL, inspired and continued to aid the author; his help is gratefully acknowledged.

Dr. Shirley Pomponi-Taylor, now of the University of Maryland, exposed test specimens and accumulated weight data at the Key Biscayne exposure site. Mr. John Keeton and Mr. John Crahan of CEL were responsible for the mix design and preparation of the concrete test specimens.

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Table 1. Materials Used and Manufacturers

Item	Description or Designation	Source
Adhesive	Super Weatherstrip Adhesive, Part No. 8003	3M Co. Adhesives, Coatings & Sealers Div. St. Paul, MN 55101
Aggregate	Ridgelite-Rocklite, 3/8"	Ceramic Lightweight Aggregate 650 S. Grand Ave. Los Angeles, CA 90017
Cable Ties	Pan-ty #PHT3H-D	Panduit Corp. Tinley Park, IL 60477
Cement	ASTM Type II	Koppers Co. Tar Products Div. Fontana, CA
Creosote	Federal Specification TT-C-645b, Class III, Creosote Oil	E. I. DuPont, de Nemours & Co. Wilmington, DE
Cu <sub>2</sub> O Nethoxychlor®	cuprous oxide, powder, 98.6% 2,2-bis(p-methoxyphenyl)-1,1-trichloroethane	B. F. Goodrich & Co. Akron, OH
Nails	6-d FSN 5315-00-889-2744	Ameron Corp. Monterey Park, CA
Nofoul®	TBTO-impregnated rubber sheeting	M & T Chemicals, Inc. Rahway, NJ 07065
Organotin Polymer	siloxane-tin polymeric coating	M & T Chemicals Inc. Rahway, NJ 07065
Sand	concrete sand (river)	
TBTO	bis(tri-n-butyltin) oxide	
TPH	triphenyltin hydroxide	

Table 2. Toxicants Employed in Antifouling Concretes

Set No.	Identification	Toxicant (Concentration) <sup>a</sup>	Remarks
1	U1, U2 C1, C2	none TBTO:creosote, 60:40 (9.0)	control
2	UT PG TB KR	none none TBTO (9.7) TBTO:creosote, 60:40 (8.8)	control control
3	V1 V2 V3 V4	TBTO (4.3) TBTO (4.3)/Cu <sub>2</sub> O (2.0) Cu <sub>2</sub> O (2.0) none	control
4	V5 V6 V7	methoxychlor (4.3) methoxychlor (4.3)/TPTH (4.8)/Cu <sub>2</sub> O (4.7) organotin polymer (unknown)	
5	NF	No-foul rubber (unknown)	

<sup>a</sup>Percent by weight of all other constituents.

Table 3. Comparison of Subjective and Objective Methods of Rating Fouling

[First Set of Specimens at Port Hueneme]

Subjective Method of Rating		
Fouling Organism	Density of Fouling <sup>a</sup>	
	Treated	Untreated
Barnacles	3,7,0,F,5,2 5,7,0,9,2,2	F,2,3,2,F,11 13,3,F,6,1,1
Bryozoa	F,F,F,H,F,M F,F,F,F,H	H,H,H,H,VH,M F,M,VH,M,M,M
Tunicates	F,F,F,0,F,F F,F,F,F,F,F	F,1,H,M,F,M F,F,F,F,F,F
Mussels	0,0,0,0,0,1 0,0,0,0,0,0	2,0,F,F,2,F F,F,F,F,F,F
Objective Method of Rating		
Condition	Mean Weight Increase <sup>b</sup> of Specimens (gm)	
	Treated	Untreated
Wet	87.0	197.2
Dry	10.12	38.54

<sup>a</sup>F = few; M = medium; H = heavy; VH = very heavy; 0 = none.  
Other numbers denote the number of organisms on a specimen.

<sup>b</sup>After 173 days.

Table 4. Weight of Accumulated Biofouling

Set No.	Toxicant	Port Hueneme		Key Biscayne	
		Weight Increase (gm)	No. of Days	Weight Increase (gm)	No. of Days
1	Control	197.2	173	---	--
	TBTO/creosote	87.0	173	---	--
2	Control	447.4	596	734.6	524
	Control	234.6	401	623.3	241
	TBTO	372.8	596	705.8	524
	TBTO/creosote	234.0	596	433.6	524
3	TBTO	71.8	153	1,320	283
	TBTO/Cu <sub>2</sub> O	61.4	153	1,331	283
	CU <sub>2</sub> O	82.5	153	1,661	283
	Control	89.1	153	1,432	283
4	Methoxychlor	110.2	153	154.4	139
	Methoxychlor/TPTH/Cu <sub>2</sub> O	57.6	153	139.7	139
	Polymeric tin	37.1	153	103.2	139
	Control	89.1	153	155.3	139
5	Organotin rubber	15.8	60	no data yet	--

Table 5. Effect of Toxicant and Toxicant Concentration  
on Larvae of Haliotis Rufescens

Exposure Time (hr, min)	Effect <sup>a</sup> of Following Toxicants for Indicated Times --								
	TBTO			TBTO/Creosote			Methoxychlor/ TPTH/Cu <sub>2</sub> O		
	2	20	200	2	20	200	2	20	200
0,10	+	+	±	+	+	±	+	+	±
1,0	±	±	±	+	±	±	+	+	+
1,50	-	-	0	±	±	±	+	+	±
2,30	-	-	0	±	±	-	+	+	±
3,30	0	0	0	±	-	-	+	+	±
5,0	0	0	0	±	-	0	+	+	-
6,45	0	0	0	-	-	0	+	+	0
24,0	0	0	0	0	0	0	±	±	0
32,0	0	0	0	0	0	0	0	0	0

<sup>a</sup> + = no visible effect

± = live, most immobile

- = cellular disassociation or abnormal development

0 = dead

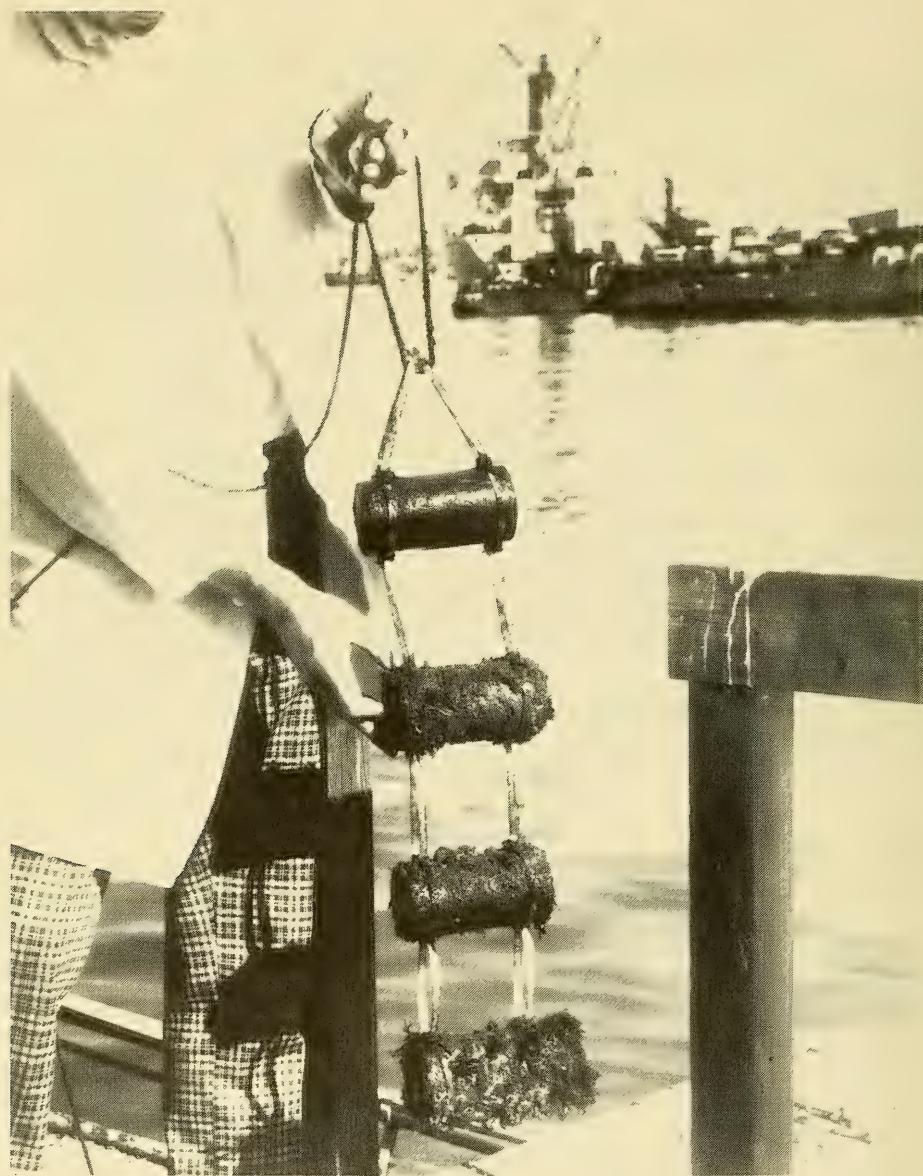


Figure 1. Cylinders prepared for exposure in ladder-like position.



Figure 2. Weight increase, Port Hueneme, set no. 2.



Figure 3. Weight increase, Key Biscayne, set no. 2.

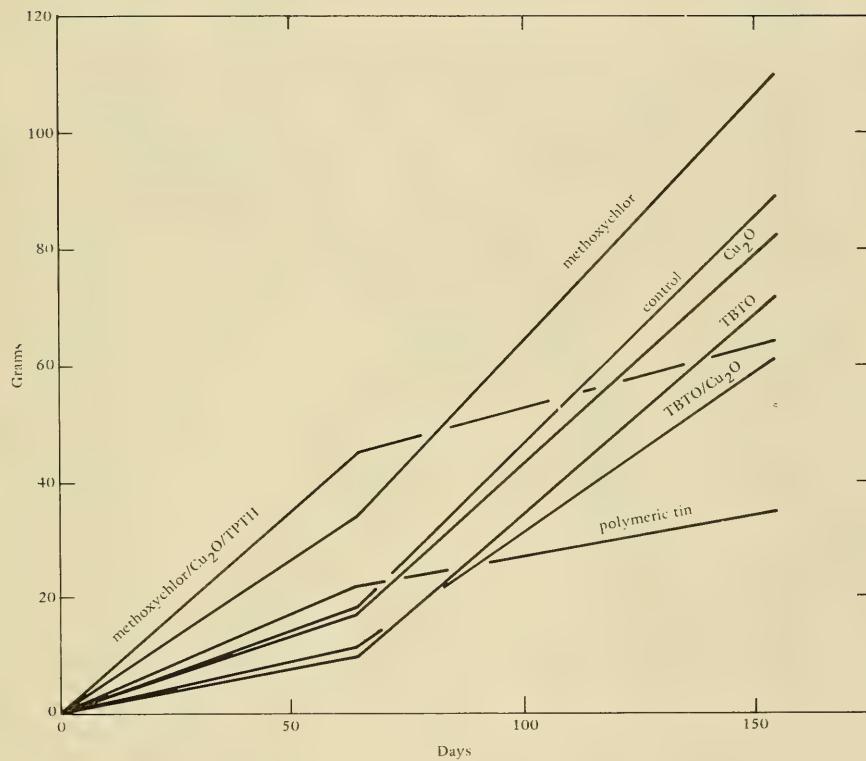


Figure 4. Weight increase, Port Hueneme, set no. 3 and 4.

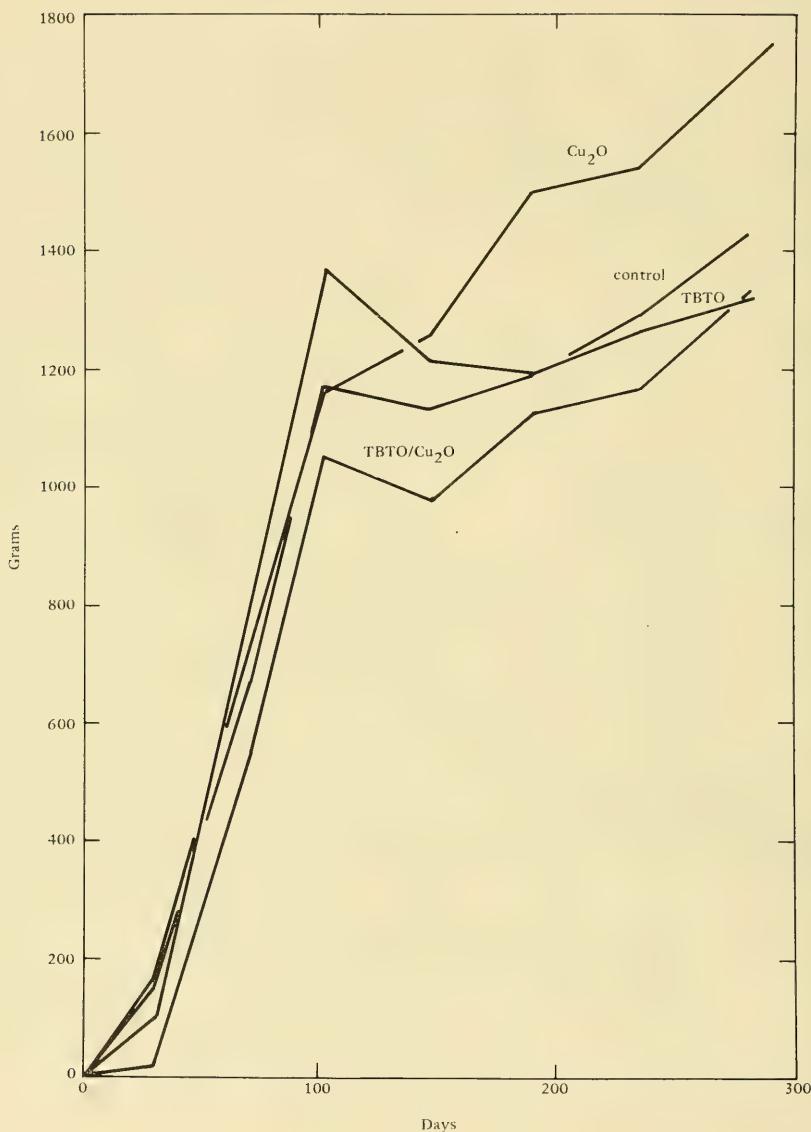


Figure 5. Weight increase, Key Biscayne, set no. 3.

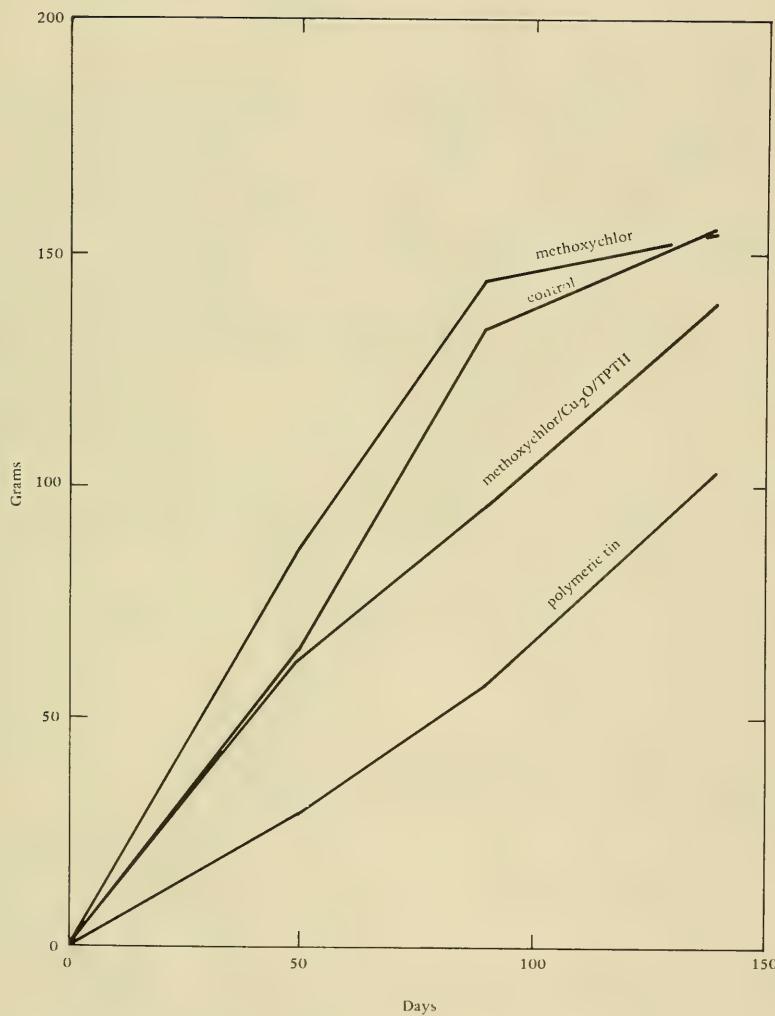


Figure 6. Weight increase, Key Biscayne, set no. 4.

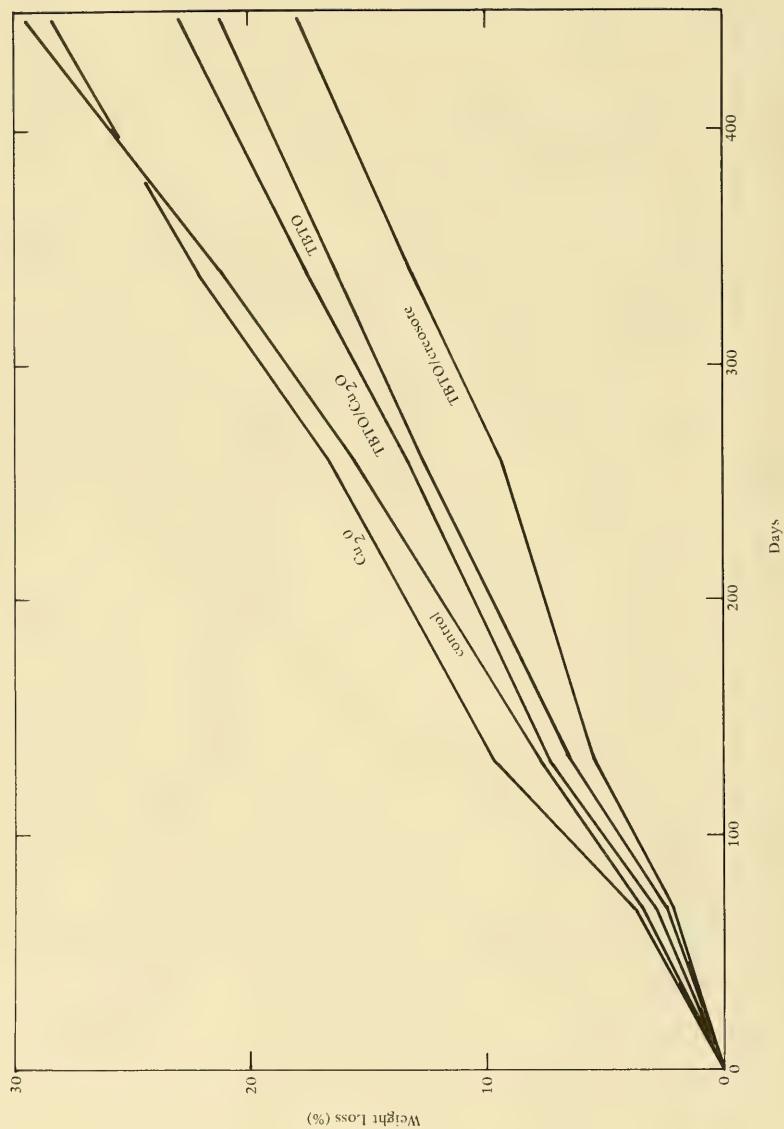


Figure 7. Effect of toxicant on weight loss of simulated reinforcing rod.

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NAS CO, Guantanamo Bay Cuba; Code 114, Alameda CA; Code 183 (Fac. Plan BR MGR); Code 18700, Brunswick ME; Code 18U (ENS P.J. Hickey), Corpus Christi TX; Code 6234 (G. Trask), Point Mugu CA; Code 70, Atlanta, Marietta GA; Code 8E, Patuxent Riv., MD; Dir. Maint. Control Div., Key West FL; Dir. Util. Div., Bermuda; ENS Buchholz, Pensacola, FL; Lakehurst, NJ; Lead. Chief Petty Offr. PW/Self Help Div, Beeville TX; OIC, CBU 417, Oak Harbor WA; PW (J. Maguire), Corpus Christi TX; PWD Maint. Cont. Dir., Fallon NV; PWD Maint. Div., New Orleans, Belle Chasse LA; PWD, Maintenance Control Dir., Bermuda; PWD, Willow Grove PA; PWO Belle Chasse, LA; PWO Chase Field Beeville, TX; PWO Key West FL; PWO Whiting Fld, Milton FL; PWO, Dallas TX; PWO, Glenview IL; PWO, Kingsville TX; PWO, Millington TN; PWO, Miramar, San Diego CA; PWO,, Moffett Field CA; ROICC Key West FL; SCE Lant Fleet Norfolk, VA; SCE Norfolk, VA; SCE, Barbers Point HI; Security Offr, Alameda CA  
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NAVCOMMAREAMSTRSTA Code W-602, Honolulu, Wahiawa HI; PWO, Norfolk VA; PWO, Wahiawa HI; SCE Unit 1 Naples Italy  
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NAVEDTRAPRODEVcen Tech. Library  
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NAVELEXSYS COM Code PME-124-61, Washington DC  
NAVENVIRHLTHCEN CO, Alexandria, VA  
NAVEODFAC Code 605, Indian Head MD  
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NAVFAC PWO, Lewes DE  
NAVFACENGCOM Code 042 Alexandria, VA; Code 043 Alexandria, VA; Code 044 Alexandria, VA; Code 0451 Alexandria, VA; Code 0453 (D. Potter) Alexandria, VA; Code 0454B Alexandria, VA; Code 046; Code 0461D (V. M. Spaulding) Alexandria, VA; Code 04B (M. Yachnis) Alexandria, VA; Code 04B3 Alexandria, VA; Code 04B5 Alexandria, VA; Code 081B Alexandria, VA; Code 100 Alexandria, VA; Code 1002B (J. Leimanis) Alexandria, VA; Code 1023 (T. D. Stevens), Alexandria VA; Code 1113 (M. Carr) Alexandria, VA; Code 1113 (T. Stevens) Alexandria, VA; Code 1113 Alexandria, VA; Morrison Yap, Caroline Is.; PC-2 Alexandria, VA; PC-2 Alexandria, VA

NAVFACENGCOM - CHES DIV. Code 101 Wash, DC; Code 102, (Wildman), Wash, DC; Code 402 (D Scheselle) Washington, DC; Code 403 (H. DeVoe) Wash, DC; Code 405 Wash, DC; Code FPO-1 Wash, DC; Contracts, ROICC, Annapolis MD; FPO-1 (Spencer) Wash, DC; FPO-1 Wash, DC

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NAVFACENGCOM - NORTH DIV. (Boretsky) Philadelphia, PA; AROICC, Brooklyn NY; CO; Code 09P (LCDR A.J. Stewart); Code 1028, RDT&ELO, Philadelphia PA; Code 111 (Castranova) Philadelphia, PA; Code 114 (A. Rhoads); Design Div. (R. Masino), Philadelphia PA; ROICC, Contracts, Crane IN

NAVFACENGCOM - PAC DIV. (Kyi) Code 101, Pearl Harbor, HI; Code 2011 Pearl Harbor, HI; Code 402, RDT&E, Pearl Harbor HI; Commander, Pearl Harbor, HI

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NAVFACENGCOM - WEST DIV. 102; 112; AROICC, Contracts, Twentynine Palms CA; Code 04B San Bruno, CA; 09P/20 San Bruno, CA; RDT&ELO Code 2011 San Bruno, CA

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Maint. Control Dir., Yorktown VA  
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NAVWPNSUPPCEN Code 09 Crane IN  
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Hueneme, CA; Code 25111 Port Hueneme, CA; Code 400, Gulfport MS; NESO Code 251 P.R. Winter Port  
Hueneme, CA; PW Engrg, Gulfport MS; PWO (Code 80) Port Hueneme, CA; PWO, Davisville RI  
NCBU 411 OIC, Norfolk VA  
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200, Guam; Code 220 Oakland, CA; Code 220.1, Norfolk VA; Code 30C, San Diego, CA; Code 400, Great  
Lakes, IL; Code 400, Oakland, CA; Code 400, Pearl Harbor, HI; Code 400, San Diego, CA; Code 420,  
Great Lakes, IL; Code 420, Oakland, CA; Code 42B (R. Pascua), Pearl Harbor HI; Code 505A (H.  
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